Characterization of Damage Accumulation in a C/SiC Composite at Elevated Temperatures

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Introduction and Motivation

This research is part of a program aimed to evaluate and demonstrate the ability of candidate CMC materials for a variety of applications in reusable launch vehicles. The life and durability of these materials in rocket and engine applications are of major concern and there is a need to develop and validate life prediction methodology. In this study, material characterization and mechanical testing was performed in order to identify the failure modes, degradation mechanisms, and progression of damage in a C/SiC composite at elevated temperatures. The motivation for this work is to provide the relevant damage information that will form the basis for the development of a physically based life prediction methodology.

Material

The material used in this study is a DuPont Lanxide C/SiC manufactured using a proprietary infiltration procedure. The fiber architecture was a simple 0/90 two dimensional weave laminate. The composite was fabricated in 8" x 8"x 0.125" panels. Test specimens, having a typical dog bone geometry, were machined using diamond tooling and re-infiltrated following machining in order to protect the newly exposed surfaces.

The typical surface condition an microstructure of the composite is shown in Figure 3. It is obvious that in the as-received condition the material exhibits a variety of inherent cracking within the matrix, fiber tows, and surface. The majority of the internal cracks seem to be contained within the [90] plies, and the majority of the surface cracks seem to be within the coating itself.

Testing

The material was characterized by performing a variety of tensile, creep, and fatigue testing. The bulk of the mechanical testing was performed at 650°C (1200°F). Limited testing was also performed at 550 °C (1022°F) and 450 °C (842°F) in order to determine the temperature regime where the environmental effect is dominant.

In order to determine the progression and accumulation of damage, a significant number of tests were interrupted prior to failure and subjected to NDE and metallographic inspection. A NDE method was used to determine the resonant frequency as a function of life of a stress rupture specimen that was tested at 650 ^{o}C and 70 MPa and periodically interrupted. Similarly, stress rupture specimens tested at 650 ^{o}C and 70 MPa were interrupted after 30min, 75min, and 150min (corresponding to 10%, 25%, and 50% of life) and sectioned for metallography. Also where applicable, particularly in the fatigue tested specimens, the stiffness was monitored as a function of life.

Results

Stress Rupture Behavior

The stress-rupture behavior at various elevated temperatures is shown in Figures 4 and 5. The strength of the composite degrades very rapidly for temperatures above 550 ^{o}C. At 650 ^{o}C and 70 MPa the life is approximately 5hrs. At 450 ^{o}C and 200 MPa on the other hand the life is significantly greater than 50 hours and furthermore there is little degradation in the residual strength. The effect of damage accumulation was evident in the stress strain behavior of an interrupted specimen. As shown in Figure 6, a decrease in stiffness occurred very early in life and the degradation continued throughout the test. Similar degradation was observed in the resonant frequency response, Figure 7. At 650 ^{o}C the applied load has very little effect on the life, this observation in conjunction with the fact that damage occurs very early in life not only suggests that the environment governs the damage process but also that the presence of inherent cracking hastens the process.

Fatigue Behavior

Low cycle fatigue testing was performed in order to determine if any synergistic process occurs in the presence of fatigue. The results are shown in Figure 8 in comparison to the stress rupture data. In general, the strain accumulation is slower, and overall longer lives are attained during fatigue. The effect of cyclic induced damage was further investigated by performing a fatigue test at much higher frequency (100 Hz). The results indicate that even though the cycles to failure increased by several orders of magnitude, the time to failure was in the same order as the lower frequency fatigue tests and stress rupture tests, Figure 9. This observation reinforces the notion that overall at 650 ^{o}C the damage accumulation is governed by environmental exposure. Fatigue damage doesn't appear be have any additional effects. Similar to the observations of stress rupture damage, the damage accumulation that was observed in the low cycle fatigue tests manifests itself in the reduction of stiffness as shown in Figure 10. The reduction of stiffness with respect to the life fraction is not only consistent for the various fatigue testing conditions but it coincides well with the creep rupture behavior. This behavior further implies that damage accumulation is environmentally driven. Most importantly, however, this behavior, if indicative of the actual damage, can serve as the basis for life prediction methodology.

Metallography

Extensive metallography and fractographic examination was performed on the failed and interrupted specimens with the primary purpose of determining the mechanisms of damage. The surface of the specimens did not reveal any additional damage aside from the already existing cracks, Figure 11. This suggests that the coated surface (i.e. the matrix) is not an overwhelming contributor to the damage process and that the damage responsible for the failure is occurring internally.

This became evident upon inspection of the metallographic sections obtained from the interrupted specimens. As seen in Figure 12, fibers near the surface are being destroyed by the oxidizing environment which enters the specimen by the myriad of inherent cracks, Figure 13. This phenomenon is also apparent on the fracture surface of the stress rupture specimens, Figure 14, where for the most part the C fibers are missing and seem to have been consumed. The carbon fibers on the tensile specimens, on the other hand, are intact. Also evident from the interrupted tests is a progression of damage. The specimen interrupted at 50% of the life show the most extensive damage while the specimen interrupted at 10% of life exhibits the least amount of damage. The fact that damage appears at a very early stage in life, and manifests itself in the form of fiber damage, gives support to the observations that stiffness degradation reflects the presence and progression of damage.

Discussion and Summary

Based on the observations of the various mechanisms present and the material behavior, the damage process of C/SiC at elevated temperatures can summarized as follows:

1. early in life the existing cracks propagate into the composite and are bridged by the fibers
2. these cracks act as pipelines for the environment, which attacks the C fibers
3. failures in the oxidized fibers results into further crack propagation and linking up of cracks
4. as more fibers are oxidized the bridging effect diminishes until the net section in one of the locally oxidized regions is insufficient to carry and transfer load

In general, the damage seems to accumulate as crack propagation, linking up of cracks, and environmental degradation of the load bearing fibers. The toughening mechanisms, although very complex and interrelated, can be categorized as bridging, multiple cracking, and crack tortuousity. These mechanisms are also manifested at various scales. For example, crack bridging can occur within a fiber tow as shown in Figures 12 and 13 where individual fibers are bridging the crack. Also bridging can occur on a larger scale, as shown in Figure 15, where the whole fiber tows are bridging cracks. Understanding the interrelationships of these mechanisms as well as their degradation due to the environment is vital to developing vital and reliable life prediction methodology.

Conclusions

1. The strength of the C/SiC CMC degrades significantly during stress rupture testing for temperatures above 550 ^{o}C, whereas below 450 ^{o}C degradation is minimal.
2. Degradation of the modulus seems to occur very early in life and continue throughout the test. Low cycle fatigue testing at the same testing conditions exhibited longer time to failure than the stress rupture testing.
3. Examination of failed and interrupted specimens indicates that the C fibers are oxidizing very rapidly at these temperatures.
4. The accumulation of damage, the failure process, and degradation of strength above 550 ^{o}C is dominated by environmental degradation of the carbon fibers.

Future Work

The damage data obtained so far has been mainly qualitative, in the future emphasis will be placed on quantifying the observed damage and correlating it with observed mechanical behavior such as resonant frequency or stiffness degradation. This will serve as the basis for developing and evaluating a life prediction model based on actual material behavior. Also, the material fatigue properties will be further evaluated at lower temperature regimes where the environment does not dominate the failure process and damage accumulation occurs by other mechanisms. This will ensure that the major environmental and fatigue damage processes are incorporated in the lifing model.

MOTIVATION

LONG TERM GOAL:

- DEVELOP, CODIFY, AND VALIDATE LIFE PREDICTION METHODOLOGY FOR CERAMIC MATRIX COMPOSITE ENGINE COMPONENTS FOR REUSABLE LAUNCH VEHICLES.

SHORT TERM GOAL:

- IDENTIFY DEGRADATION MECHANISMS, DAMAGE PROGRESSION, AND FAILURE MODES TO SUPPORT THE DEVELOPMENT OF LIFING MODELS.

APPROACH

- CHARACTERIZE MECHANICAL BEHAVIOR OF 2-D WEAVE DuPONT LANXIDE C\SiC CMC (TENSILE, CREEP, AND FATIGUE PROPERTIES).
- IDENTIFY DEGRADATION MECHANISMS, DAMAGE PROGRESSION, AND FAILURE MODES IN THE CMC.
- DEVELOP LIFE MODELS BASED UPON CAPTURING AND MODELING THE PHYSICS OF THE DAMAGE ACCUMULATION PROCESSES.

Fig. 1

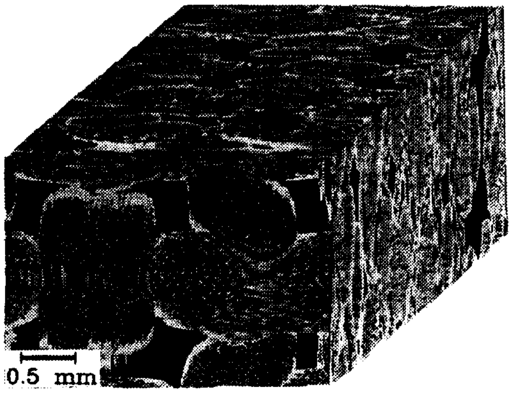
EXPERIMENTAL PROCEDURE

- ELEVATED TEMPERATURE TENSILE, CREEP, AND FATIGUE TESTS
- TEST TEMPERATURE: 650 °C (1200 °F), 550 °C (1022 °F), AND 450 °C (842 °F)
- STIFFNESS DATA MONITORED AND STORED THROUGHOUT THE TEST
- NDE INSPECTION PERFORMED ON SELECTED SPECIMENS (SPECIMENS WERE SUBJECTED TO INTERRUPTED TESTING AND REPEATED INSPECTION)
- FRACTOGRAPHIC AND METALLOGRAPHIC CHARACTERIZATION OF FAILED AND INTERRUPTED SPECIMENS

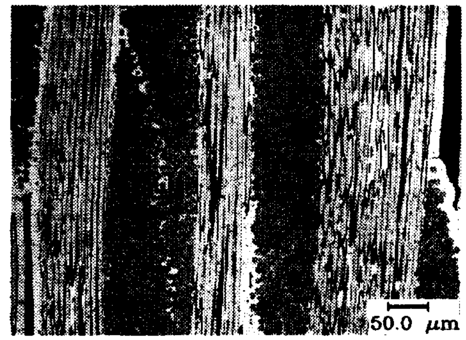
Fig. 2

MATERIAL

DUPONT LANXIDE
C/SiC CMC
2-D WEAVE LAMINATE



CMC HAS INHERENT CRACKING DUE TO FABRICATION



INTERNAL CRACKING CONTAINED WITHIN [90°] PLIES

SURFACE CRACKING CONTAINED WITHIN SURFACE MATRIX COATING

Fig. 3

Stress-Rupture Data for DuPont C/SiC

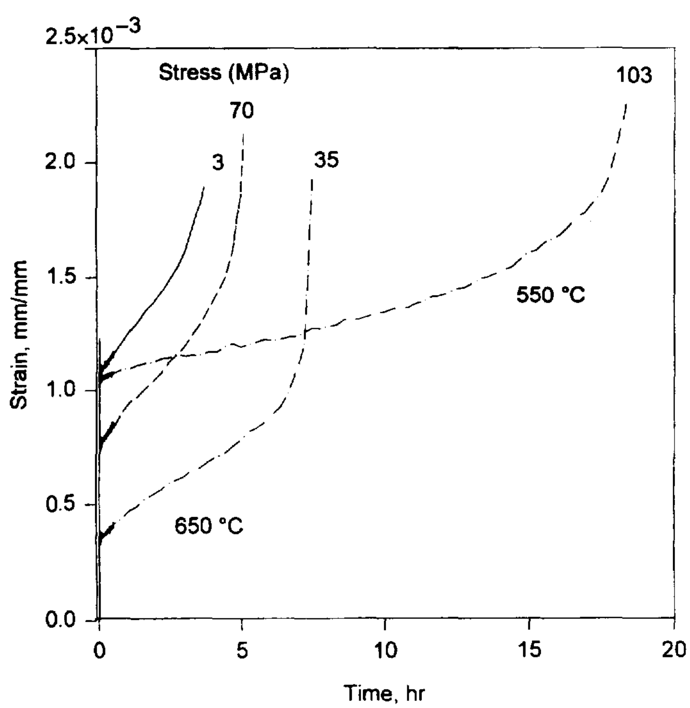
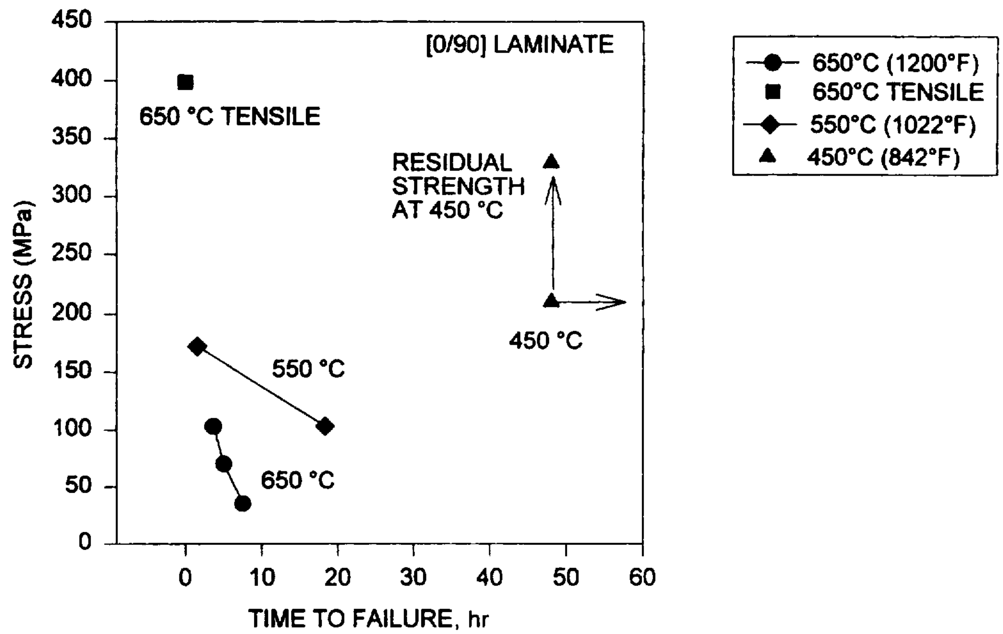


Fig. 4

STRESS-RUPTURE OF C/SiC CMC AT VARIOUS TEMPERATURES



- STRENGTH OF CMC DEGRADES RAPIDLY ABOVE 550°C
- AT 450°C DEGRADATION IS MINIMAL

Fig. 5

STIFFNESS DEGRADATION AS A FUNCTION OF LIFE FRACTION DURING STRESS RUPTURE

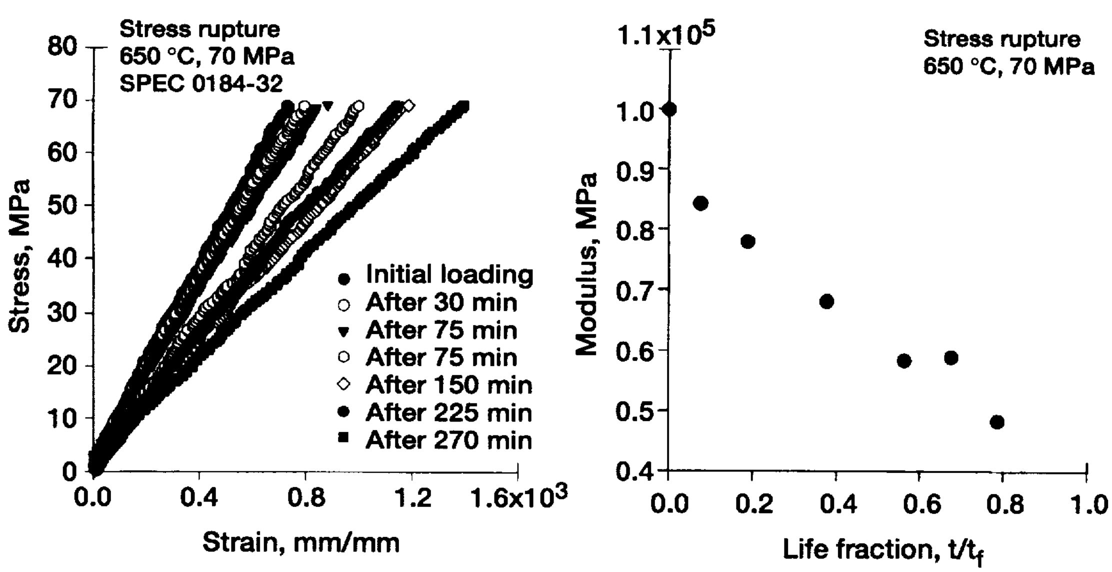
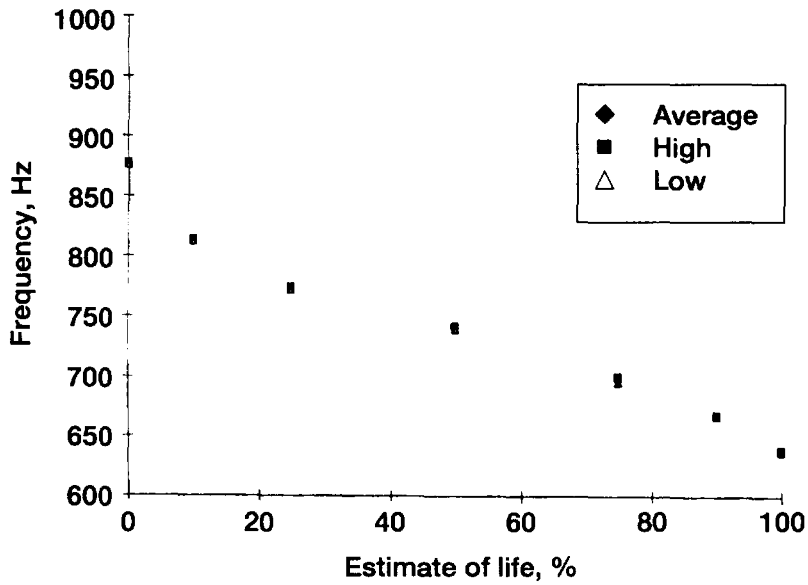


Fig. 6

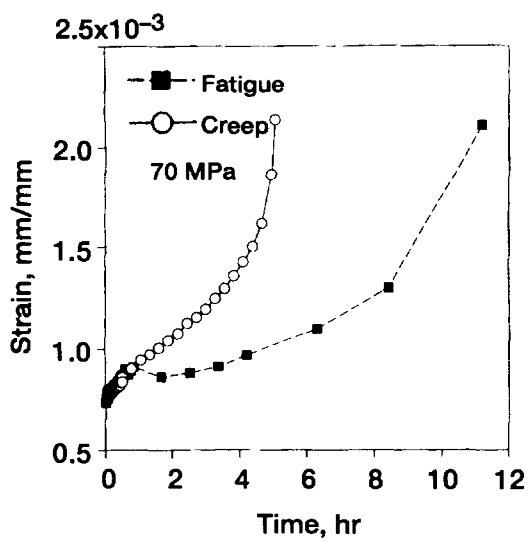
RESONANT FREQUENCY RESULTS FOR INTERRUPTED 650 °C STRESS-RUPTURE TEST

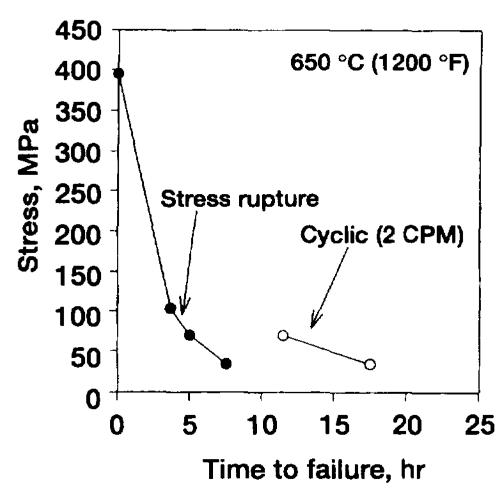


- VERY SENSITIVE AND HIGHLY REPEATABLE NDE TECHNIQUE
- CAPTURES CHANGE IN RESPONSE IN THE VERY EARLY STAGES

Fig. 7

STRESS-RUPTURE AND FATIGUE COMPARISONS AT 650 °C

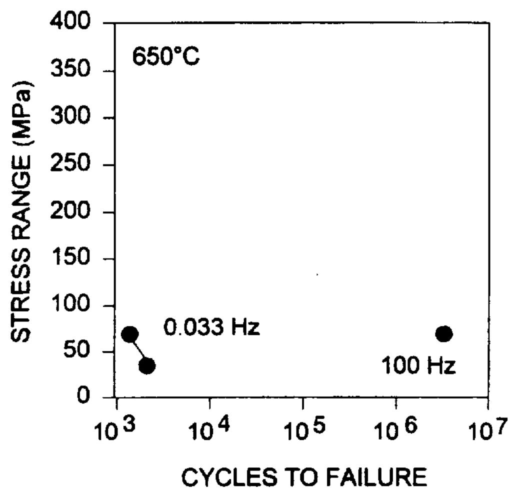


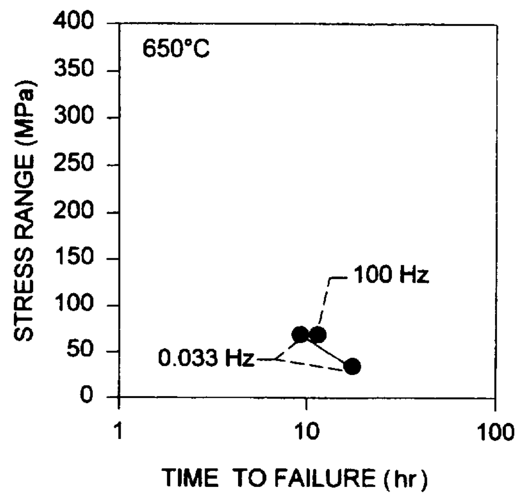


- STRAIN ACCUMULATES SLOWER DURING FATIGUE
- FATIGUE TESTS RESULT IN LONGER LIVES

Fig. 8

EFFECT OF CYCLIC FREQUENCY ON LCF RESULTS



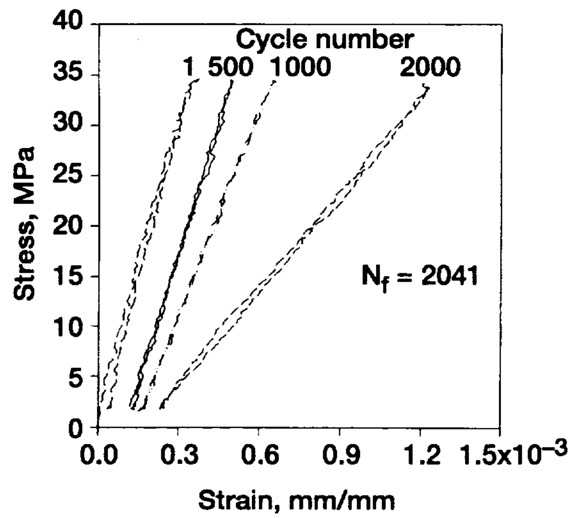


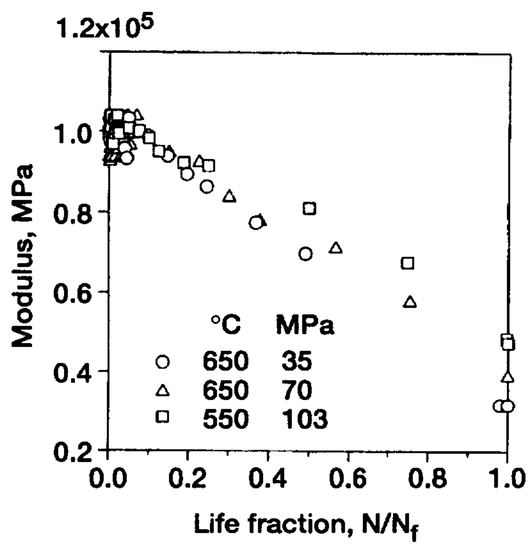
- CYCLIC FAILURE IS GOVERNED BY TIME AT TEMPERATURE

- ENVIRONMENTAL DEGRADATION CONTROLS FAILURE AT 650°C

Fig. 9

STIFFNESS DEGRADATION AS A FUNCTION OF LIFE FRACTION DURING LOW CYCLE FATIGUE



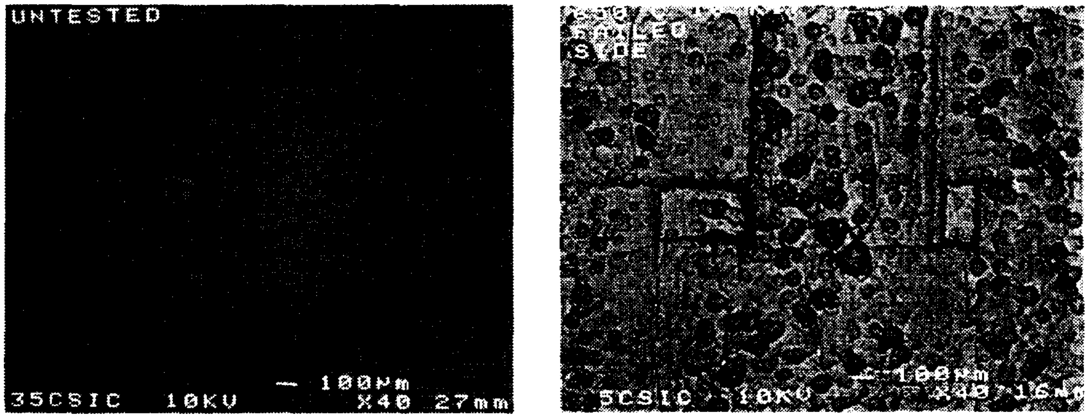


PHENOMENA COULD BE BASIS FOR LIFE PREDICTION MODEL

Fig. 10

SPECIMEN SURFACE BEFORE AND AFTER TESTING

AS–FABRICATED CONDITION | **TESTED AT 650 ^{o}C, 70 MPa**



NETWORK OF FINE CRACKS PRESENT IN THE AS FABRICATED CONDITION

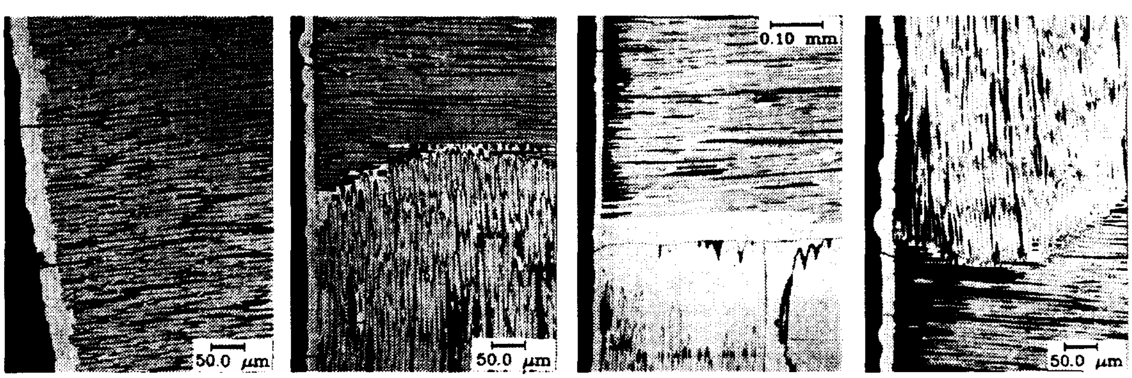
DENSITY AND APPEARANCE OF PRE–EXISTING CRACKS DO NOT CHANGE DURING TESTING

DAMAGE MUST BE INTERNAL

Fig. 11

DAMAGE ACCUMULATION AT 650 ^{o}C AND 70 MPa

AS FABRICATED | **10% OF LIFE** | **25% OF LIFE** | **50% OF LIFE**



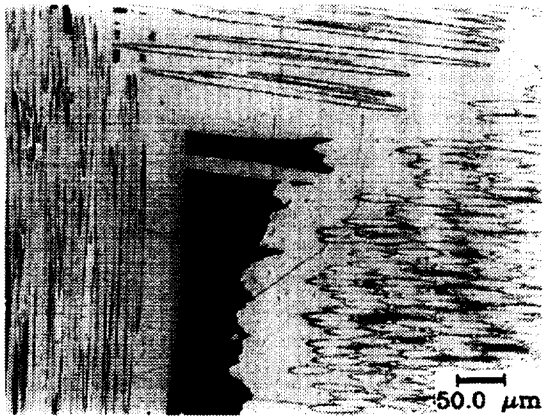
0 MIN | **30 MIN** | **75 MIN** | **150 MIN**

PRE–EXISTING CRACKS ACT AS PIPELINES FOR THE ENVIRONMENT

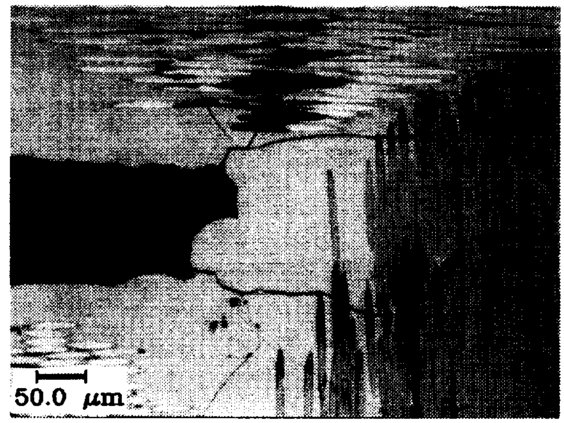
Fig. 12

ENVIRONMENTAL DAMAGE AT 650 °C

AS-FABRICATED CONDITION



TESTED AT 650 °C, 70 MPa



CRACKS INITIALLY ARE BRIDGED BY THE FIBERS

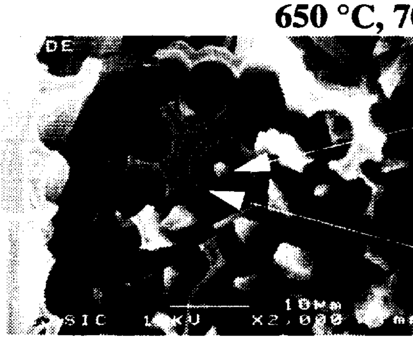
EXPOSURE AT 650 °C RESULTS IN FIBER OXIDATION AND LOSS OF BRIDGING

Fig. 13

FRACTOGRAPHY

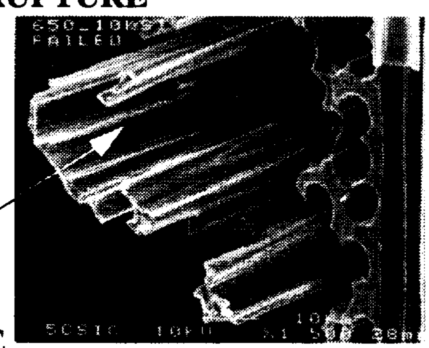
*** CARBON FIBERS SEVERELY OXIDIZED DURING STRESS RUPTURE TEST**
*** MOSTLY CRACKED SiC MATRIX LEFT NEAR SPECIMEN SURFACE**

650 °C, 70 MPa STRESS RUPTURE

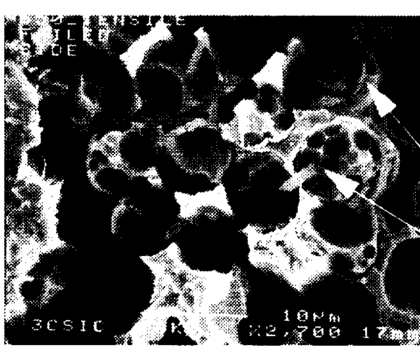


C FIBER

SiC MATRIX



650 °C TENSILE



SiC MATRIX

C FIBER

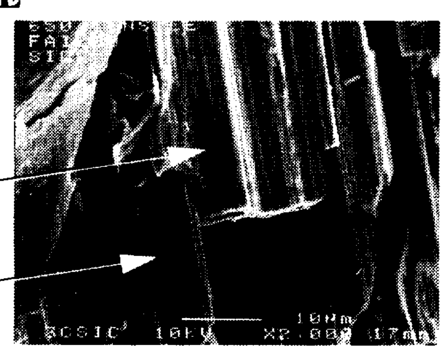


Fig. 14

DAMAGE PROGRESSION IN C/SiC AT 650 ^{o}C

1) REGULAR ARRAYS OF OXIDIZED CRACKS FORM VERY EARLY IN LIFE
2) CRACKS ARE BRIDGED BY VARIOUS METHODS
3) OXIDATION OF THE FIBERS DESTROYS THE BRIDGING, RESULTING IN FAILURE

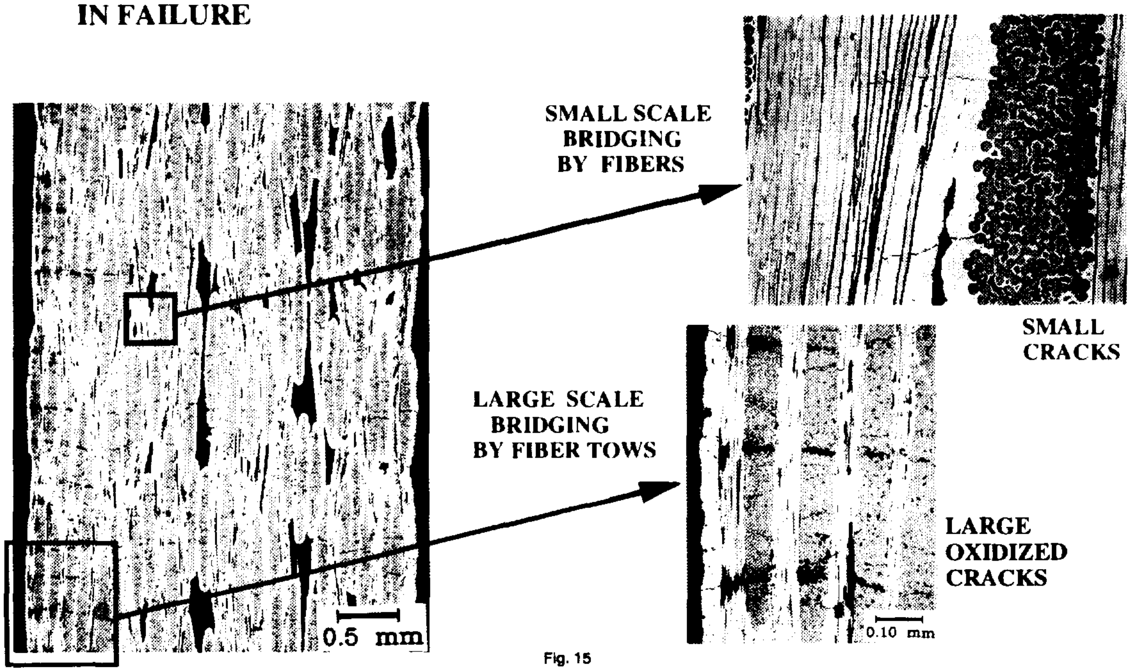


Fig. 15

SUMMARY AND CONCLUSIONS

1. THE STRENGTH OF THE C/SiC CMC DEGRADES SIGNIFICANTLY DURING STRESS RUPTURE TESTING FOR TEMPERATURES ABOVE 550 ^{o}C, WHEREAS BELOW 450 ^{o}C DEGRADATION IS MINIMAL.

2. LOW CYCLE FATIGUE TESTING AT THE SAME TESTING CONDITIONS PRODUCED <u>LONGER TIME TO FAILURE</u> THAN THE STRESS RUPTURE TESTING.

3. DEGRADATION OF THE MODULUS SEEMS TO OCCUR VERY EARLY IN LIFE AND CONTINUE THROUGHOUT THE TEST FOR BOTH STRESS RUPTURE AND FATIGUE LOADING.

4. FRACTOGRAPHIC EXAMINATION OF FAILED SPECIMENS AND METALLOGRAPHIC EXAMINATION OF SPECIMENS FROM INTERRUPTED TESTS INDICATES THAT THE CARBON FIBERS ARE OXIDIZING VERY RAPIDLY AT THESE TEMPERATURES.

5. THE ACCUMULATION OF DAMAGE, THE FAILURE PROCESS, AND DEGRADATION OF STRENGTH ABOVE 550 ^{o}C IS DOMINATED BY ENVIRONMENTAL DEGRADATION OF THE CARBON FIBERS.

Fig. 16

FUTURE WORK

- QUANTIFY AND CORRELATE OBSERVED DAMAGE AND STIFFNESS DEGRADATION TO ESTABLISH A BASIS FOR A DAMAGE ACCUMULATION MODEL

- INVESTIGATE FATIGUE MECHANISMS AND DAMAGE ACCUMULATION AT LOWER TEMPERATURE REGIMES WHERE ENVIRONMENTAL EFFECTS ARE NOT PRESENT

- INCORPORATE BOTH ENVIRONMENTAL AND FATIGUE DAMAGE MECHANISMS INTO A PHYSICALLY BASED LIFE PREDICTION MODEL

Fig. 17